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(71) Applicant  
Dynamit Nobel  
Aktiengesellschaft,  
521, Troisdorf near  
Cologne,  
Germany

(72) Inventors  
Franz Blaschke,  
Norbert Vollkommer,  
Peter Gebauer,  
Werner Schmidt,  
Herbert Klinkenberg

(74) Agents  
Haseltine Lake & Co.,  
Hazlitt House,  
28, Southampton  
Buildings,  
Chancery Lane,  
London,  
WC2A 1AT

(54) Improvements in or relating to  
polytetramethylene terephthalate-  
shaped bodies

(57) Unreinforced or reinforced  
polytetramethylene terephthalate-  
shaped bodies is stabilised with  
triglycidyl isocyanurate and/or a

bisoxazoline, as well as optionally a  
phenolic or a phosphitic antioxidant.  
Shaped bodies can be produced from  
such stabilised polymer, especially by  
injection moulding, with reduction of  
the viscosity of the polymer and  
deterioration of the material  
properties of the pure polymer itself to  
any significant extent.



between 12% and 17% (in comparison with more than 180% for pure PTMT, the PTMT worked up in like manner without flame protecting agent). For reinforced PTMT the drop in mechanical strength values caused by the presence of a flame protecting agent is likewise very disadvantageous, there being a fall in the impact resistance from 36 to 25—30 KJ/m<sup>2</sup> and in notched bar impact resistance from 9 to 10 to 5—7 KJ/m<sup>2</sup> in contrast to pure PTMT.

Moreover, because such flame protected PTMT-shaped bodies have a lower stability during working and use than reinforced PTMT or reinforced PTMT which is not flame protected there is much scope for problems in the event of for example a breakdown in the injection moulding working, too long residence in the injection moulding machine, unsuitable machine adjustment or use of temperatures higher or lower than those ascertained as optimal. In such cases, there is immediately obtained a further reduction in molecular weight and products with further deteriorated mechanical properties are obtained.

Such flame protected, but mechanically deteriorated products will generally be unusable. In the use test with storage of the extrudates for 3 days at 150°C in air it is observed that the impact resistance can drop to below 50 KJ/m<sup>2</sup> at 100% break, the notched bar impact resistance to below 3 KJ/m<sup>2</sup> and the elongation at break to below 10%. Moreover, a complete embrittlement of the product is obtained under the test conditions.

It has previously been proposed to stem the reduction of the molecular weight degradation by PTMT-shaped bodies by adding to the PTMT a polymeric epoxy resin of equivalent weight 300 to 2000 g/mol in amounts between 0.5 and 20% (German Offenlegungsschrift 28 21 292). In this way there is achieved an improvement in molecular weight, but  $\eta_{red}$  is still only 1.21—1.41 dl/g in the injection moulding in contrast to  $\eta_{red}$ =1.53 dl/g in pure PTMT and this is insufficient for the compounding.

$$\left(\eta_{red} = \frac{\eta - \eta}{\eta} \cdot 1/c\right)$$

wherein  $\eta$ =viscosity of the solvent phenol/o-Dichlorobenzene=60/40,  $\eta$ =viscosity of the solution and c=concentration of the solution in g/100 ml).

Moreover, the suitability for use, the results obtained in the use test carried out on the final extrudate and the  $\eta_{red}$  value in the extrudate also remain unsatisfactory if, according to German Offenlegungsschrift No. P 28 21 292, resins, PTMT, polytetraabromobenzyl acrylate and Sb<sub>2</sub>O<sub>3</sub> are employed together (see Table 6, Comparative Examples H and J).

There exists therefore the need also to provide conveniently an unreinforced or reinforced PTMT which contains only a small amount of flame protecting agent and of impact resisting agent of the aforementioned co- or graft polymeric type, and which after extrusion and injection moulding possesses comparable mechanical and special properties to injection moulded products formed of pure PTMT or pure PTMT provided only with reinforcing fillers as additives.

More particularly, in general, there is a need to be able to provide PTMT simultaneously with a number of special types of additives to impart reinforcement, impact resistance and flame protection to it, while avoiding the degradation or strong deterioration of the good material properties of PTMT which usually takes place when additives are incorporated therein.

According to the present invention, there is provided a polytetramethyleneterephthalate shaped body which is formed of polytetramethylene-terephthalate and contains triglycidyl isocyanurate and/or a bisoxazoline as stabiliser.

It has been found the use of quite specific stabilisers, namely triglycidyl isocyanurate and/or a bisoxazoline, for example the phenylene or tetramethylene isoxazoline leads to suppression of the aforementioned undesirable phenomena. The effect of these stabilisers is particularly surprising since stabilisers of other type do not hinder the degradation of PTMT and do not lead to maintenance of the special material properties of PTMT.

The present invention will now be described in detail with reference to four types of additives which may be present in polytetramethylene terephthalate shaped bodies, namely (1) reinforcing fillers, (2) impacting resisting agents, (3) phenolic or phosphitic antioxidants and (4) flame protecting agents, which may be used alone or in combination.

Thus this invention is applicable, not only to unreinforced PTMT shaped bodies but to reinforced PTMT-shaped bodies, especially such bodies when reinforced with glass fibres. Preferably from 15 to 80% by weight of reinforcing fillers, related to PTMT are present.

Glass fibres have the advantage of possessing a so-called active reinforcing effect. Otherwise it is possible to use, for example asbestos fibres or organic polymer fibres, for example high melting polyester, polyurethane, polysulphone, aliphatic or aromatic polyamide fibres as reinforcing fillers.

Moreover this invention provides shaped polytetramethylene terephthalate bodies with contents of co or graft polymers of ethylene and vinyl acetate or co- or graft polymers based on acrylic acid or methacrylic acid esters as impact resisting agents. The impact resisting agents in the form of the co- or graft polymers are preferably added in amounts of 5 to 30, more preferably 12 to 25% by weight related to PTMT.

This invention also provides PTMT shaped bodies which contain flame protecting agents based on polymerised bromine-containing acrylates or methacrylates and optionally also the aforementioned impact resisting agents.

The stabilisers surprisingly cause the molecular weight drop on working in the extruder and in the subsequent injection mould to be reduced very much; even when using small amounts of the additives, the degradation of PTMT on standing in the ambient atmosphere is can be suitably influenced. The largely hindered degradation of PTMT on use of the shaped bodies is valuable. The hydrolysis of PTMT as a result of the stabilisers almost completely held back. The impact resisting agents are only effective as a result of the stabilisers added according to the invention. Moreover, as a result of the presence of the stabilisers flame protecting agents based on high polymerised bromine-containing acrylates which are solid materials,

The addition of ethylene/vinyl acetate co- or graft polymers is especially valuable taken together with reinforcing fillers. Of especial value are such PTMT bodies reinforced with glass fibres consisting of:

- a) 45—83, preferably 55—71 parts by weight PTMT with a reduced specific viscosity  $\eta_{red}$  of 1.2—2.2 dl/g preferably 1.5—1.9 dl/g. ( $\eta_{red}$  is defined as previously indicated).
- b) 15—40, preferably 25—35 parts by weight glass fibres,
- c) 2—20, preferably 4—10 parts by weight of ethylene/vinyl acetate copolymers or graft polymers (EVA)
- d) 3—30, preferably 10—20% by weight of co- or graft polymers based on acrylic acid esters or methacrylic acid esters.

Furthermore, unreinforced shaped bodies can also be produced, containing 85 to 98% by weight PTMT and 2 to 15% by weight EVA and/or 3 to 30% by weight flame protecting agent which is a polymerised bromine-containing acrylate or methacrylate.

It is surprising that the aforementioned comparatively small amounts of EVA are sufficient. No further increased impact resistance can be achieved with larger amounts of EVA.

Suitable copolymers of ethylene and vinylacetate are those with contents of vinyl acetate of 2 to 65% by weight, preferably 5 to 50% by weight. The copolymers are known materials and can be produced by copolymerisation in suspension or in bulk. They are generally used in powder form.

By the terms co- or graft polymers based on acrylic acid esters or methacrylic acid esters are understood those polymers in which at least 5% by weight, preferably 1 to 6 carbon atoms in the alcohol group of acid and/or methacrylic acid ester with 1 to 10, preferably 1 to 6 carbon atoms in the alcohol group of the ester, are present. The second component of the such polymers is preferably another of the indicated acrylic or methacrylic acid esters of a different type of polymerisable olefinic compound such as for example an olefin, acrylonitrile or styrene. In the case of ternary polymers, there will be a further one of the indicated monomers—in the case of graft polymers a graft substrate of two of the components or of butadiene-styrene and preferably an acrylic acid or methacrylic acid ester is present as grafting agent. Preferred are mixed polymers of 20 to 50, preferably 30 to 40% by weight of an acrylic or methacrylic acid methyl or ethyl ester and a further acrylic or methacrylic acid propyl to butyl ester as well as ter- and graft polymers of 5 to 40, preferably 10 to 30% by weight of an acrylic or methacrylic acid ester, especially a methyl ester, and 40 to 80, preferably 50 to 70% by weight butadiene and 5 to 35, preferably 20 to 30% by weight styrene.

The combined effect of the stabilisers and the impact resisting co- or graft polymers is surprising for in the absence of the claimed stabilisers, co- or graft polymers (comparative examples A to G) achieve practically no improved impact or notched bar impact resistance in comparison with the basic material. In addition to improving the notched bar resistance, the addition of the stabilisers achieves an increase in the elongation at break and the Youngs modulus.

Furthermore the stabilisers affect favourably the temperature stressing to which the material can be subjected during working and increases in particular the working range, that it makes possible the use of a greater temperature range in the extruder or in the injection moulding machine without harming and loss of the composition. Stabilisers of other type do not achieve this synergistic composite effect.

The stabiliser(s) is/are preferably used in an amount of 0.1 to 2.5, more preferably 0.1 to 1.5% by weight based on the weight of PTMT. Insofar as triglycidyl isocyanurate is used alone, an upper limit of 1.0% by weight is preferably placed on the amount added; insofar as p- or m-bisoxazoline is used, its quantity preferably amounts to not more than 1.5% by weight.

It is preferred to add as well a phosphitic antioxidant and a phenolic antioxidant additional to triglycidyl isocyanurate and/or the bisoxazoline. In general, it is feasible for only one of the two antioxidants to be added.

Phosphitic antioxidants are phosphites of at least tri-functional alcohols, especially of pentaerythritol, or for example of trimethylolpropane or possibly even glycerol, wherein one or two of the OH-groups of the phosphoric acid are esterified with a fatty alcohol residue, especially a  $C_8$  to  $C_{24}$  fatty acid residue, preferably the stearyl residue or the decyl residue. The preferred alcohol is pentaerythritol.

Phenolic antioxidants will generally be of the type in which there is a sterically hindered phenolic

group, especially one containing a t-butyl group in the o-position to the phenolic OH-group. Examples of such anti-oxidants are many. Preferred examples are, however, 2,2'-thiodiethylbis-[3-(3,5-di-tert.butyl-4-hydroxyphenyl)]-propionate and 1,3,5-trimethyl-2,4,6-tri-(4-hydroxy-3,5-di-tert.butyl)benzene.

Triglycidyl isocyanurate and one of the bisoxazolines achieves, alone or together, a decisive improvement in the mechanical properties, although the added amount is small. The reduced viscosity  $\eta_{red}$  of the PTMT in the injection mould after extrusion and working is surprisingly equally high or even higher than that of the pure PTMT starting material.

Moreover the two above-indicated types of antioxidant improve very much the scope for working, the stability in use for example as measured in the indicated use test, as well as the colour of the product; that is discolouration is avoided.

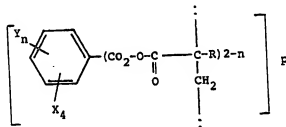
In general, when using antioxidants in the production of shaped bodies, it is preferred to use from 0.02 to 0.5, more preferably 0.05 to 0.3% by weight 2,2'-thiodiethyl-bis-[3-(3,5-di-tert.butyl-4-hydroxyphenyl)]-propionate and/or 0.05 to 0.5, more preferably 0.1 to 0.3% by weight distearyl hydroxytritol phosphite, especially with the aforementioned amounts of the stabilisers. When an impact-resisting agent and/or a flame protecting agent is present the amount of the 2,2'-thiodiethylbis-[3-(3,5-di-tert.butyl-4-hydroxyphenyl)]-propionate is preferably 0.02 to 0.3, more preferably 0.05 to 0.2% by weight. These percentages are based on the weight of PTMT.

In the production of reinforced PTMT use is preferably made of PTMT with  $\eta_{red}=1.2$  to 1.3, since here a PTMT of  $\eta_{red}=1.6$  to 1.7, which is not only further condensed, but is also more expensive, brings no essential advantage. For unreinforced PTMT, in contrast, PTMT of  $\eta_{red}=1.6$  and higher is preferred.

Reference has already been made herein to the application of this invention to unreinforced or reinforced PTMT-shaped bodies containing flame protecting agents which are polymerised bromine-containing acrylates or methacrylates.

While some acceptable losses in impact resistance, notched impact resistance and elongation at break, are obtained when using such flame protecting agents, it is possible as a result of stabilisation according to the invention to produce a satisfactory unreinforced flame protected PTMT based on PTMT—1.2, that is  $\eta_{red}=1.2$  (Examples 21 and 22) when, without stabilisation or with addition of bisphenol-A-diglycidyl ethers, a complete injection moulding is obtained which is a completely unusable material for constructional uses.

As polymeric, bromine-containing acrylates, there can be used homo- and copolymers containing units of the formula



wherein X signifies preferably bromine, optionally with proportions of chlorine, p the degree of polymerisation,  $n=0$  or 1, Y denotes Br or  $-\text{CH}_3$  and R hydrogen, methyl, ethyl or propyl. The production of such polymers is known from German Auslegungsschrift 25 27 802. It is also possible to use polymers of such type obtained from the monomers of German Offenlegungsschrift 28 00 020, polymers and copolymers according to German Offenlegungsschrift 26 12 843 as well as copolymers according to German Offenlegungsschrift 26 48 969, and the corresponding bromine-containing acrylates of benzylic alcohols or xylene glycols. It is preferred, however, to use the especially simply preparable PBB-PA, that is polymers of pentabromobenzyl acrylate, optionally with proportions of tribromo or tetrabromobenzyl acrylates. In addition to the aforementioned acrylates, the corresponding methacrylates can be used, on the basis of comparability, if where in the examples PBB-PA is given as a flame protective agent, a correspondingly good flame protecting effect is achievable with the other known bromine-containing polymeric acrylates, especially if the bromine content approximately equals that of PBB-PA. The flame protecting agent is preferably used in amounts of 5 to 20% by weight related PTMT.

$\text{Sb}_2\text{O}_3$  can be used as synergist to the flame protecting agent in amounts of from 1 to 10% by weight related to PTMT.

In testing for the extent of flame protection achieved, as in the examples which follow, the 1.6 mm flat rods required according to UL 94 (Underwriters Laboratories, subject UL 94) and LOI (lowest oxygen index, that is the  $\text{O}_2$  proportion in an  $\text{O}_2/\text{N}_2$  mixture in volume % which acts just self-extinguishingly) are produced on an injection moulding machine made by the ARBURG COMPANY.

The general mechanical properties were determined according to the following test

55 specifications:

	Impact resistance	DIN 53 453	
	Notched bar impact resistance	DIN 53 454	
	Bending resistance (limit of bending stress)	DIN 53 452	
5	Tensile strength (stretching stress)	DIN 53 455	5
	Resistance to tearing	DIN 53 455	
	Elongation at break Thermal non-deformability according to ISO/R 75; A+B	DIN 53 461	

- 10 Table 6 to be set out hereinafter shows the high stability in use of the stabilised flame protected PTMT compositions according to the invention in comparison with the state of the art as expressed by Comparative Examples H (without stabiliser) and J (with an epoxide, EP GY 250 of CIBA GEIGY as stabiliser), testing being carried out in the ambient atmosphere for storage over different times and with subsequent measurement of the changes with time of impact resistance, notched bar impact resistance and elongation at break.

- 15 In the production of shaped bodies or compositions embodying this invention, the dry constituents thereof are mixed well and co-extruded by means of an R 45-single screw extruder of the REIFENHAUSER COMPANY in a temperature region of 240 to 260°C. The extruded strand is withdrawn through a water bath and granulated. After the drying of the granulate, test rods are produced on an injection moulding machine of the firm KRAUSS-MAFFEI in a universal shape which is necessary for the testing of the mechanical and thermal properties. Operating in this way it has quite surprisingly been shown that the shaped bodies, irrespective of whether one or more of the optionally present components reinforcing filler, impact resisting agent, and flame protective means are present, are producible merely by co-extrusion and are obtained completely homogeneous for the purpose of working up by for example injection moulding. Accordingly, in the simplest conceivable manner, each constituent can be supplied to the extruder as powder or granulate using a suitable metering arrangement.

- The stabilisers and antioxidants and  $Sb_2O_3$  may merely be dusted or sprinkled onto one of the components or are mixed therewith, although that is also not always essential.
- 30 Such a co-extrusion is quite unusual since generally through mixing gives rise to some problems and overall addition is then essentially by premixing or double extrusion. In fact the co-extrusion which can be carried out in producing the shaped bodies of this invention reduces essentially the degradation of the PTMT.

- Thus, this invention also provides a process for the production of PTMT shaped bodies according to this invention, which comprises continuously supplying to an extruder polytetramethylene terephthalate, triglycidyl isocyanurate and/or a bisoxazoline and optionally one or more further components of the body, which component(s) is/are chosen from a said reinforcing filler, said impact resisting agents and said polymerised bromine-containing acrylate or methacrylate flame protecting agent, which component(s) is/are supplied separately from one another and separately from the polytetramethylene terephthalate, the triglycidyl isocyanurate and/or bisoxazoline and, if used, the phosphitic or phenolic antioxidant and/or  $Sb_2O_3$  being simultaneously supplied continuously and separately from one another to the extruder either together with the polytetramethylene terephthalate or one or more of said components or as materials supplied separately from any other constituent(s) of the body, and co-extruding said constituents in the extruder.

- 45 This procedure may, in principle, be used when producing reinforced and unreinforced PTMT shaped bodies, which even contain no impact resisting co- or graft polymers of the indicated type or flame protecting agent, and the only additives are for achieving stabilisation with the indicated stabiliser system of triglycidyl isocyanurate and/or bisoxazoline and the optional addition of phosphitic antioxidants and/or phenolic antioxidants which is itself of high value.

- 50 The amounts of the stabilisers and antioxidants added are therefore essentially the same in general, irrespective of what further constituents are present, and in the preferred region which has already been given.

- The degradation which usually takes place as a result of the essentially high working temperatures of 230 to 270°C in the production of the shaped body by extrusion and then working and shaping by for example injection moulding, with the consequence of lower molecular weight of viscosities and strongly reduced properties in use is prevented essentially by the use of the aforementioned stabiliser system without which cross-linking of the PTMT occurs.
- 55 In contrast to when using unstabilised PTMT, the stabiliser system achieves a preservation of important and valuable mechanical properties of the PTMT starting material, such as for example the impact resistance and/or notched bar impact resistance. This widens the scope for working of PTMT. In particular, the improvement in the melt stability of the PTMT under working conditions means that thermal deterioration of the PTMT as a result of disturbances and delays in the working or prolongation of the heating time is minimised. The thermal-oxidative degradation during working and consequential discolouration of the product are therefore largely reduced.

In particular, the retention of stability in use as a result of the stabilisation system has been shown in a test in air at 180°C. This test shows that the impact resistance is maintained at "not broken" with unstabilised PTMT for only a few days, whereas with stabilised samples after 10 days the impact resistance has the value not broken and the notched bar impact resistance is likewise reduced only slightly after about 10 days. In a similar manner, a PTMT shaped body reinforced with glass fibres exhibits only a small reduction in the impact resistance and notched bar impact resistance when subjected over many days to thermal stressing.

A further important effect is the stabilisation with respect to hydrolysis which can be checked by hot water storage of injection mouldings at 80°C. Here, the test shows that the original impact resistance, notched bar impact resistance and elongation at break with unstabilised samples is only maintained for about 4 weeks, whereas the original values of impact resistance and notched bar impact resistance are maintained for about 12 weeks in the hydrolysis test carried out on the stabilised samples. This applies in particular for unreinforced PTMT; the effect of stabilisation of PTMT with glass fibre reinforcement is not quite so impressive.

The following Examples illustrate this invention. In the examples all parts are on a weight basis.

#### Examples 1 to 22

In each of these examples, and the associated Comparative Examples, to produce the indicated PTMT composition, the dry components were premixed well and co-extruded through a single screw extruder with a screw diameter of 45 mm in the temperature range 240 to 280°C. The extruded strand was withdrawn through a water bath and granulated. After the drying of the granulate, the injection mouldings required for the testing of the mechanical and thermal properties were produced on an injection moulding machine of the firm KRAUSS-MAFFEI in a universal mould.

Examples 1 to 3 and Comparative Example A use ethylene-vinyl acetate copolymer (EVA) with 18% by weight vinyl acetate. Examples 4 and 5 as well as Comparative Examples B to D the same but with 7.5% by weight vinyl acetate. GF denotes glass fibre. The impact resisting graft polymer M<sub>1</sub> consists of about 20% by weight methylmethacrylate, about 60% by weight butadiene and about 20% by weight styrene. The mixed polymer M<sub>2</sub> consists of about 65% by weight n-butyl acrylate and about 35% by weight methyl methacrylate.

Moreover, in the Examples, the reference letters I) to VII) have the following means:

- I) 2,2'-Thiodiethylbis-3-(3,5-ditert.butyl-4-hydroxyphenyl)-propionate
- II) Diisobutyl pentaerythritol phosphite
- III) Triglycidyl isocyanurate
- IV) p-phenylenebisoxazoline
- V)  $\eta_{red} = \eta_{sp/c}$  (1% in Pheno/O-Dichlorobenzene=60/40)
- VI) Araldite GY 250 CIBA-GEIGY (Epoxide-equivalent weight about 180—190 g/mol Epoxide)
- VII) Epikote 1001 of SHELL COMPANY

The PTMT samples used in the examples are denoted by reference letters as follows:

For PTMT-A,  $\eta_{red} = 1.76$  dl/g  
 PTMT-B,  $\eta_{red} = 1.67$  dl/g  
 PTMT-C,  $\eta_{red} = 1.6$  dl/g  
 PTMT-D,  $\eta_{red} = 1.2$  dl/g

Other abbreviations appearing in the examples are  
 B=broken  
 NB=not broken

Table 1

		Example 1 64 parts PTMT-A 30 parts GF 6 parts EVA Stabiliser: 0.1 part I) +0.2 part II) +0.4 part III)	Example 2 60 parts PTMT-A 30 parts GF 10 parts EVA Stabiliser: 0.2 part I) +0.2 part II) +0.5 part III)	Comparative Example A 64 parts PTMT-A 30 parts GF 8 parts EVA without Stabiliser
5		KJ/m <sup>2</sup> 46	49	36
10	Impact Resistance			
	Notched bar impact resistance:			
	23°C	KJ/m <sup>2</sup> 24	25	14
	-20°C	KJ/m <sup>2</sup> 18	20	11
	-40°C	KJ/m <sup>2</sup> 17	18	10
		N/mm <sup>2</sup> 124	119	110
15	Tear resistance			
	Youngs Modulus (Drawing test)	N/mm <sup>2</sup> 9400	8800	7600
		N/mm <sup>2</sup> 190	182	173
	Limit of bending test			
20	Thermal non-deformability			
	according to	°C 186	178	172
	ISO/R75: A	°C >200	>200	>200
	ISO/R75: B	N/mm <sup>2</sup> 151	—	—
	Ball pressure hardness			

Table 1 (continued)

		Example 3 66 parts PTMT-A 30 parts GF 4 parts EVA Stabiliser: 0.1 part I) +0.2 part II) +0.6 part III)	Example 4 64 parts PTMT-A 30 parts GF 6 parts EVA Stabiliser: 0.1 part I) +0.2 part II) +0.4 part III)	Example 5 60 parts PTMT-A 30 parts GF 10 parts EVA Stabiliser: 0.2 part I) +0.3 part II) +0.4 part III)
25		KJ/m <sup>2</sup> 44	47	48
30	Impact Resistance			
	Notched bar impact resistance:			
	23°C	KJ/m <sup>2</sup> 19	23	26
	-20°C	KJ/m <sup>2</sup> 16	17	18
	-40°C	KJ/m <sup>2</sup> 14	15	17
		N/mm <sup>2</sup> 129	126	115
35	Tear resistance			
	Youngs Modulus (Drawing test)	N/mm <sup>2</sup> 9800	9700	8600
		N/mm <sup>2</sup> 187	196	182
	Limit of bending test			
	Thermal non-deformability			
40	according to	°C 192	190	—
	ISO/R75: A	°C >200	>200	>200
	ISO/R75: B	N/mm <sup>2</sup> 164	159	149
45	Ball pressure hardness			



Table 1 (continued)

		Comparative Example B 60 parts PTMT-A 30 parts GF 10 parts EVA Without stabiliser	Comparative Example C 70 parts PTMT-A 30 parts GF Without stabiliser	Comparative Example D 70 parts PTMT-A 30 parts GF Stabiliser: 0.1 part I) +0.2 part II) +0.5 part III) 37
5		KJ/m <sup>2</sup> 38	35	
10	Impact Resistance			
	Notched bar impact resistance:			
	23°C	KJ/m <sup>2</sup> 13	9—10	9—11
	-20°C	KJ/m <sup>2</sup> 12	—	—
	-40°C	KJ/m <sup>2</sup> 11	8—9	8—10
		N/mm <sup>2</sup> 112	130	133
15	Tear resistance			
	Youngs Modulus (Drawing test)	N/mm <sup>2</sup> 6700	10000	10500
	Limit of bending test	N/mm <sup>2</sup> 164	190	200
	Thermal non-deformability			
20	according to	°C 168	200	—
	ISO/R75: A	°C >200	>200	—
	ISO/R75: B	N/mm <sup>2</sup> —	175	—
	Ball pressure hardness			

Table 2

		Example 6 85 parts PTMT-A 15 parts M <sub>1</sub> Stabiliser: 0.1 part I) +0.2 part II) +0.5 part III) without break	Example 7 88 parts PTMT-A 12 parts M <sub>1</sub> Stabiliser: 0.1 part I) +0.2 part II) +0.6 part III) without break	Example 8 82 parts PTMT-A 18 parts M <sub>1</sub> Stabiliser: 0.2 part I) +0.3 part II) +0.4 part III) without break
25		KJ/m <sup>2</sup>		
30	Impact resistance			
	Notched bar impact resistance:			
	23°C	KJ/m <sup>2</sup> 44	32	47
	-20°C	KJ/m <sup>2</sup> 18	14	19
	-40°C	KJ/m <sup>2</sup> 14	11	16
		N/mm <sup>2</sup> 43	45	42
	Tensile strength	N/mm <sup>2</sup> 30	32	32
	Resistance to tear	% 125	107	140
	Elongation at break	N/mm <sup>2</sup> 69	72	65
40	Bending resistance			
	Youngs Modulus (from bending experiment)	N/mm <sup>2</sup> 1900	1980	1850
	Thermal non-deformability			
	according to	°C 55	56	51
45	ISO/R75: A	°C 151	154	143
	ISO/R75: B			

**Example 9**  
85 parts PTMT-A  
15 parts M<sub>1</sub>  
Stabiliser:

	Table 2 (continued)		
	Example 9 85 parts PTMT-A 15 parts M <sub>1</sub> Stabiliser: 0.1 part I) +0.2 part II) +0.4 part IV) without break	Example 10 82 parts PTMT-A 18 parts M <sub>1</sub> Stabiliser: 0.1 part I) +0.2 part II) +0.5 part IV) without break	
Impact resistance		48	
Notched bar impact resistance:		—	
23°C	KJ/m <sup>2</sup>	17	
-20°C	KJ/m <sup>2</sup>	13	
-40°C	N/mm <sup>2</sup>	44	
Tensile strength	N/mm <sup>2</sup>	30	
Resistance to tear	N/mm <sup>2</sup>	130	
Elongation at break	%	64	
Bending resistance	N/mm <sup>2</sup>	1700	
Youngs Modulus (from bending experiment)	N/mm <sup>2</sup>	—	
Thermal non-deformability according to ISO/R75: A	°C	—	
ISO/R75: B	°C	—	

**Comparative  
Example E  
85 parts PTMT-A  
15 parts M<sub>1</sub>**

		Table 2 (continued)		
		Comparative Example E 85 parts PTMT-A 15 parts M <sub>1</sub> without Stabiliser without break	Comparative Example F 82 parts PTMT-A 18 parts M <sub>1</sub> without Stabiliser without break	
25				30
30	Impact resistance	KJ/m <sup>2</sup>		
	Notched bar impact resistance:			
	23°C	KJ/m <sup>2</sup>	12	16
	-20°C	KJ/m <sup>2</sup>	8	10
	-40°C	KJ/m <sup>2</sup>	6	7
35	Tensile strength	N/mm <sup>2</sup>	42	39
	Resistance to tear	N/mm <sup>2</sup>	28	27
	Elongation at break	%	35	30
	Bending resistance	N/mm <sup>2</sup>	57	55
40	Youngs Modulus (from bending experiment)	N/mm <sup>2</sup>	1500	1450
	Thermal non-deformability			
	according to	°C	53	—
	ISO/R75: A	°C	145	—
45	ISO/R75: B			

**Example 11**  
85 parts PTMT-B  
15 parts M<sub>2</sub>  
Stabiliser:

**Example 12**  
80 parts PTMT-B  
20 parts M<sub>2</sub>  
Stabiliser:

5			Stability 0.1 part I) +0.2 part II) +0.4 part IV) without break	0.1 part I) +0.2 part II) +0.6 part IV) without break	
10	Impact resistance	KJ/m <sup>2</sup>			10
	Notched bar impact resistance:				
	23°C	KJ/m <sup>2</sup>	23	26	
	—20°C	KJ/m <sup>2</sup>	9	11	
	—40°C	KJ/m <sup>2</sup>	7	8	
	Tensile strength	N/mm <sup>2</sup>	43	41	15
15	Resistance to tear	N/mm <sup>2</sup>	29	28	
	Elongation at break	%	60	65	
	Bending resistance	N/mm <sup>2</sup>	70	57	
	Youngs Modulus (from bending experiment)	N/mm <sup>2</sup>	1950	1850	20
20	Thermal non-deformability				
	according to	°C	54	52	
	ISO/R75: A	°C	148	137	
	ISO/R75: B				

**Example 13**  
85 parts PTMT-B  
15 parts M<sub>2</sub>  
Stabiliser:

**Comparative  
Example G**  
85 parts PTMT-B  
15 parts  $M_2$

30		0.2 part I) +0.2 part II) +0.5 part III) without break	1.0 part I) +0.5 part II) without stabiliser without break	30
	Impact resistance	KJ/m <sup>2</sup>		
	Notched bar impact resistance:			
	23°C	KJ/m <sup>2</sup>	21	14
35	-20°C	KJ/m <sup>2</sup>	—	—
	-40°C	KJ/m <sup>2</sup>	—	5
	Tensile strength	N/mm <sup>2</sup>	44	44
	Resistance to tear	N/mm <sup>2</sup>	30	29
	Elongation at break	%	50	26
40	Bending resistance	N/mm <sup>2</sup>	71	56
	Youngs Modulus (from bending experiment)	N/mm <sup>2</sup>	1970	1800
	Thermal non-deformability			
	according to	°C	—	55
45	ISO/R75: A	°C	—	136
	ISO/R75: B			

**Table 4**  
**Unreinforced PTMT-compositions**

		<i>Example 14</i> 86 parts PTMT-C 10 parts PBB-PA 4 $Sb_2O_3$ Stabiliser: 0.1 part I) +0.2 part II) +0.5 part III)	<i>Example 15</i> 86 parts PTMT-C 10 parts PBB-PA 4 parts $Sb_2O_3$ Stabiliser: 0.1 part I) +0.1 part II) +0.2 part III)	<i>Example 16</i> 86 parts PTMT-C 10 parts PBB-PA 4 parts $Sb_2O_3$ Stabiliser: 0.1 part I) +0.2 part II) +1.0 part IV) all NB
5				
10	Impact resistance	KJ/m <sup>2</sup>	5.8	5.4
	Notched bar impact resistance	N/mm <sup>2</sup>	95	93
	Bending resistance			
	Youngs Modulus (from bending test)	N/mm <sup>2</sup>	2600	2500
15	Tensile strength	N/mm <sup>2</sup>	59	60
	Tear resistance	N/mm <sup>2</sup>	34	37
	Elongation at break	%	120—140	40—60
	Thermal non-deformability			
20	according to ISO/R75: A	°C	68	62
	ISO/R75: B	°C	167	161
	Flame protection according to UL 94		VO	VO
25	" LOI	%	30—31	30—31
	$\eta_{red}$ (V) (Granulate)		1.81	1.76
	$\eta_{red}$ (V) (Injection moulding)		1.87	1.69

**Table 4 (continued)**  
**Unreinforced PTMT-compositions**

		<i>Example 17</i> 86 parts PTMT-C 10 parts PBB-PA 4 parts $Sb_2O_3$ Stabiliser: 0.1 part I) +0.1 part II) +0.7 part IV) all NB	<i>Example 18</i> 86 parts PTMT-D 10 parts PBB-PA 4 parts $Sb_2O_3$ Stabiliser: 0.1 part I) +0.2 part II) +0.5 part III) 8NB:2B all NB	<i>Example 19</i> 86 parts PTMT-D 10 parts PBB-PA 4 parts $Sb_2O_3$ Stabiliser: 0.1 part I) +0.2 part II) +1.2 part IV) 6NB:4B all NB
30				
35	Impact resistance	KJ/m <sup>2</sup>	5.2	4.0
40	Notched bar impact resistance:	N/mm <sup>2</sup>	92	94
	Bending resistance			
	Youngs Modulus (from bending test)	N/mm <sup>2</sup>	2500	2700
	Tensile strength	N/mm <sup>2</sup>	61	62
45	Tear resistance	N/mm <sup>2</sup>	34	56
	Elongation at break	%	60—80	10—20
	Thermal non-deformability			
	according to ISO/R75: A	°C	59	57
50	ISO/R75: B	°C	163	164
	Flame protection according to UL 94		VO	VO
	" LOI	%	30—31	30—31
	$\eta_{red}$ (V) (Granulate)		1.77	1.21
55	$\eta_{red}$ (V) (Injection moulding)		1.69	1.23

Table 4 (continued)  
Unreinforced PTMT-compositions

		Unreinforced PTMT Composite		Comparative Example H	Comparative Example J	
				86 parts PTMT-C 10 parts PBB-PA 4 parts Sb <sub>2</sub> O <sub>3</sub> Without stabiliser	86 parts PTMT-C 10 parts PBB-PA 4 parts Sb <sub>2</sub> O <sub>3</sub> Stabiliser: 3 parts VJ 8NB:2B	
5						5
10	Impact resistance					10
	Notched bar impact resistance:	KJ/m <sup>2</sup>	4.1	4.0		
	Bending resistance	N/mm <sup>2</sup>	93	95		
	Youngs Modulus (from bending test)	N/mm <sup>2</sup>	2600	2500		
15	Tensile strength	N/mm <sup>2</sup>	58	62		15
	Tear resistance	N/mm <sup>2</sup>	32	36		
	Elongation at break	%	10—20	10—20		
	Thermal non-deformability according to ISO/R75: A	°C	61	—		20
20	ISO/R75: B	°C	165	—		
	Flame protection according to UL 94		VO	VO		
	LOI	%	30—31	30—31		
25	$\eta_{red}$ (V) (Granulate)		1.65	1.68		25
	$\eta_{red}$ (V) (Injection moulding)		1.54	1.58		

Table 4 (continued)  
Unreinforced PTMT-compositions

			Comparative Example K		Comparative Example L		
			86 parts PTMT-C 10 parts PBB-PA 4 parts Sb <sub>2</sub> O <sub>3</sub> Stabiliser: 4 parts VIIJ		Pure PTMT-C all NB		
30				9NB:1B			30
	Impact resistance	KJ/m <sup>2</sup>		4.2		5.5	
35	Notched bar impact resistance:	N/mm <sup>2</sup>		96		86	35
	Bending resistance						
40	Youngs Modulus (from bending test)	N/mm <sup>2</sup>		2600		2400	40
	Tensile strength	N/mm <sup>2</sup>		61		55	
	Tear resistance	N/mm <sup>2</sup>		35		34	
	Elongation at break	%		10—20		182	
45	Thermal non-deformability according to ISO/R75: A	°C		—		66	45
	ISO/R75: B	°C		—		170	
	Flame protection according to UL 94			VO		not resistant	
50	" " LOI	%		30—31		—	50
	η <sub>red</sub> (V) (Granulate)			1.66		1.76	
	η <sub>red</sub> (V) (Injection moulding)			1.52		1.67	

Table 5  
GF-reinforced PTMT-compositions

		Example 20	Example 21	Example 22
		58 parts PTMT-D	58 parts PTMT-D	58 parts PTMT-D
		30 parts GF	30 parts GF	30 parts GF
		8 parts PBB-PA	8 parts PBB-PA	8 parts PBB-PA
		4 parts Sb <sub>2</sub> O <sub>3</sub>	4 parts Sb <sub>2</sub> O <sub>3</sub>	4 parts Sb <sub>2</sub> O <sub>3</sub>
		Stabiliser:	Stabiliser:	Stabiliser:
		0.1 part I)	0.1 part I)	0.1 part I)
		+0.2 part II)	+0.2 part II)	+0.1 part II)
		+0.7 part III)	+1.0 part IV)	+0.5 part III)
5				
	KJ/m <sup>2</sup>	36	33	35
10	Impact resistance	9—10	9—10	10—11
	Notched bar impact resistance:			
	KJ/m <sup>2</sup>	220	210	220
	Bending resistance	140	135	145
15	Tensile strength			
	Thermal non-deformability			
	according to			
	ISO/R75: A	°C	198	194
	ISO/R75: B	°C	>200	>200
20	Flame protection		VO	VO
	according to UL 94			
	" LOI	%	29.5—30.5	—
	$\eta_{red}$ (V) (Granulate)	1.31	1.31	1.67
	$\eta_{red}$ (V) (Injection			
25	moulding)	1.29	1.27	1.59

Table 5 (continued)  
GF-reinforced PTMT-compositions

		Comparative Example M	Comparative Example N	Comparative Example O
		58 parts PTMT-D	58 parts PTMT-D	70 parts PTMT-D
		30 parts GF	30 parts GF	30 parts GF
		8 parts PBB-PA	8 parts PBB-PA	
		4 parts Sb <sub>2</sub> O <sub>3</sub>	4 parts Sb <sub>2</sub> O <sub>3</sub>	
		without	Stabiliser:	
		stabiliser	4 parts VI)	
30				
	KJ/m <sup>2</sup>	26	28	35
35	Impact resistance	5—7	5—7	9—10
	Notched bar impact resistance:			
	KJ/m <sup>2</sup>	190	200	220
	Bending resistance	133	136	130
	Tensile strength			
40	Thermal non-deformability			
	according to			
	ISO/R75: A	°C	186	200
	ISO/R75: B	°C	>200	>200
	Flame protection		VO	not resistant
45	according to UL 94			
	" LOI	%	1.18	1.21
	$\eta_{red}$ (V) (Granulate)			
	$\eta_{red}$ (V) (Injection	0.94	0.98	1.06
	moulding)			

**Table 6**  
Change of impact resistance, notched bar impact resistance and elongation at break with time on storage of injection mouldings at 150°C

Days	Example 14	Example 16	Comparative Example H	Comparative Example J
	Impact resistance			
0	10NB	10NB	8NB/2B	8NB:2B
3	10NB	10NB	10B	4NB:6B
5	10NB	10NB	10B	10NB
7	10NB	9NB:1B	10B	10NB
10	8NB:2B	9NB:1B	10B	10NB
15	5NB:5B	6NB:4B	10B	10NB

**Table 6 (continued)**

Days	Example 14	Example 16	Comparative Example H	Comparative Example J
	Notched bar impact resistance			
0	5.8	6.0	4.1	4.0
3	5.4	4.7	3.1	3.5
5	5.0	5.0	2.9	2.8
7	5.1	4.8	2.0	2.3
10	3.7	3.6	—	—
15	3.6	3.4	—	—

**Table 6 (continued)**

Days	Example 14	Example 16	Comparative Example H	Comparative Example J
	Elongation at break (%)			
0	125	110	12	15
3	94	70	9	11
5	70	58	5	6
7	50	37	3	3
10	40	30	—	—
15	36	26	—	—

**Example 23**

An unreinforced PTMT ( $\eta_{red}=1.76$  dl/g) was dusted with 0.1% (I), 0.2% (II) and 0.1% (III), the percentages being based on the weight of PTMT, and extruded by co-extrusion through a single screw extruder with the temperature profile of 230 to 255°C. Then standard small rods were produced on the injection moulding machine. The standard small rods were stored under the air atmosphere at 180°C. The variation of impact resistance and notched bar impact resistance with time in comparison with unstabilised samples is shown from the following table.

**Table 7**

Days	Impact resistance (KJ/m <sup>2</sup> )		Notched bar impact resistance (KJ/m <sup>2</sup> )	
	unstabilised	stabilised	unstabilised	stabilised
0	without break	without break	5.1	5.5
1	28	without break	4.2	5.4
3	—	without break	2.6	4.6
5	—	without break	2.0	3.7
7	—	without break	1.8	3.8
10	—	without break	—	4.2
15	—	40	—	4.0

In corresponding manner, PTMT with 30% by weight glass fibres and 0.1% by weight (I), 0.2% by weight (II) and 0.2% by weight (III), the percentages being based on the weight of PTMT, was provided and tested.

Table 8

Days	Impact resistance (KJ/m <sup>2</sup> )		Notched bar impact resistance (KJ/m <sup>2</sup> )	
	unstabilised	stabilised	unstabilised	stabilised
0	40	47	10	13
3	31	45	9	11
5	25	46	7	11
9	18	43	5.5	9.5

A corresponding PTMT with  $\eta_{red}=1.6$  dl/g was stabilised against hydrolysis by addition of 0.1% by weight (I), 0.2% by weight (II) and 1.2% by weight (IV), based on the weight of PTMT and tested for resistance to hydrolysis by heat storage of the injection mouldings at 80°C.

This test showed that the original impact resistance after the working was retained in the hot water test for about 12 weeks, whereas the original impact resistance with unstabilised samples was only retained for 4 weeks. In addition, the good notched bar impact resistance and elongation values were maintained in the test for 10 to 12 weeks. This points to a trouble free behaviour in use. In contrast, the unstabilised samples only maintained satisfactory notched bar impact resistance and elongation at break, which would allow their trouble-free use, for about 3 to 4 weeks.

Table 9

Weeks	Impact resistance (KJ/m <sup>2</sup> )		Notched bar impact resistance (KJ/m <sup>2</sup> )		Elongation at break (%)	
	unstabilised	stabilised	unstabilised	stabilised	unstabilised	stabilised
0	without break	without break	5.2	6.0	190	225
2	without break	without break	4.8	5.9	30	136
4	without break	without break	4.5	5.4	13	80
6	90	without break	1.6	4.5	8	50
8	10	without break	0.9	4.8	1	34
10	—	without break	—	4.5	—	24
12	—	without break	—	4.3	—	18
14	—	28	—	1.9	—	16
16	—	12	—	1.4	—	12

#### Claims

1. A polytetramethylene-terephthalate shaped body which is formed of polytetramethylene-terephthalate and contains triglycidyl isocyanurate and/or a bisoxazoline as stabiliser.
2. A body as claimed in Claim 1, in which the polytetramethylene terephthalate is reinforced.
3. A body as claimed in Claim 2, in which the polytetramethylene terephthalate is glass-fibre reinforced.
4. A body as claimed in Claim 2 or 3, which contains from 15 to 80% by weight of a reinforcing filler related to the polytetramethylene terephthalate.
5. A body as claimed in any one of the preceding claims, which additionally contains a phosphitic anti-oxidant and/or a phenolic antioxidant.
6. A body as claimed in Claim 5, wherein the phosphitic antioxidant is a phosphite of an at least trifunctional alcohol wherein one or two of the OH-groups of the phosphoric acid moiety are esterified with a fatty alcohol residue.
7. A body as claimed in Claim 6, wherein the alcohol is pentaerythritol and the fatty alcohol contains from 8 to 24 carbon atoms.
8. A body as claimed in Claim 7, wherein the alcohol is distearyl pentaerythritylphosphite.
9. A body as claimed in any one of Claims 5 to 8, wherein the phenolic antioxidant is 2,2'-thiodiethylbis-[3-(3,5-di-tert.butyl-4-hydroxyphenyl)]-propionate or 1,3,5-trimethyl-2,4,6-tri-(4-hydroxy-3,5-di-tert.butyl)benzene.
10. A body as claimed in any one of the preceding claims, which contains from 0.1 to 2.5% by weight, based on the amount of polymethylene terephthalate, of triglycidyl isocyanurate and/or bisoxazoline.
11. A body as claimed in Claim 10, which contains from 0.1 to 1.5% by weight, based on the amount of polymethylene terephthalate, of triglycidyl isocyanurate and/or bisoxazoline.
12. A body as claimed in Claim 10 or 11 which contains from 0.02 to 0.5% by weight of 2,2'-thiodiethylbis[3-(3,5-di-tert.butyl-4-hydroxyphenyl)]-propionate and/or from 0.05 to 0.5% by weight distearyl pentaerythritylphosphite.
13. A body as claimed in Claim 12, which contains from 0.05 to 0.3% by weight of 2,2'-



thiodiethylbis[3-(3,5-ditert.butyl-4-hydroxyphenyl)]-propionate and/or from 0.05 to 0.3% by weight distearyl pentaerythritylphosphite.

14. A body as claimed in any one of Claims 1 to 11 which additionally contains a co- or a graft polymer based on an acrylic acid or methacrylic ester as impact-resisting agent.

15. A body as claimed in any one of Claims 1 to 11, which additionally contains a co-polymer of ethylene and vinyl acetate as impact resisting agent.

16. A body as claimed in any one of Claims 1 to 11, which additionally contains a graft polymer of ethylene and vinyl acetate as impact resisting agent.

17. A body as claimed in Claim 15 or 16, in which the impact resisting agent contains from 2 to 65% by weight vinyl acetate.

18. A body as claimed in Claim 17, in which the impact resisting agent contains from 5 to 50% by weight vinyl acetate.

19. A body as claimed in Claim 14, wherein the impact resisting agent is a polymer of 20 to 50% by weight of an acrylic or methacrylic acid methyl or ethyl ester, together with additionally an acrylic or methacrylic acid  $C_3$  or  $C_4$  alkyl ester.

20. A body as claimed in Claim 14, wherein the impact resisting agent is a polymer of 5 to 40% by weight of an acrylic or methacrylic acid ester, 40 to 80% by weight butadiene and 5 to 35% by weight styrene, the amounts of the respective monomers to total 100%.

21. A body as claimed in any one of Claims 14 to 20, which contains the impact resisting agent in an amount of from 5 to 30% by weight of the polytetramethylene terephthalate.

22. A body as claimed in Claim 21, which contains the impact resisting agent in an amount of from 12 to 25% by weight of the polytetramethylene terephthalate.

23. A body as claimed in any one of Claims 1 to 11 and 14 to 22, which additionally contains a polymer of a bromine-containing acrylate or methacrylate as flame protecting agent.

24. A body as claimed in Claim 23, wherein the flame-protecting agent is a polymer of pentabromobenzyl acrylate.

25. A body as claimed in Claim 23 or 24, which contains from 5 to 20% by weight of the flame-protecting agent, related to the weight of polytetramethylene terephthalate.

26. A body as claimed in Claim 24 or 25, which additionally contains  $Sb_2O_3$  as synergist for the flame protecting agent.

27. A body as claimed in Claim 26, which contains from 1 to 10% by weight of  $Sb_2O_3$ , related to the weight of polytetramethylene terephthalate.

28. A body as claimed in any one of claims 14 to 27, which contains from 0.02 to 0.3% by weight of 2,2'-thio-diethylbis[3-(3,5-ditert.butyl-4-hydroxy-phenyl)]-propionate and/or 0.05 to 0.5% by weight of distearyl pentaerythritylphosphite.

29. A body as claimed in Claim 28, which contains from 0.05 to 0.2% by weight of 2,2'-thiodiethylbis[3-(3,5-ditert.butyl-4-hydroxyphenyl)]-propionate and/or 0.1 to 0.3% by weight of distearyl pentaerythritylphosphite.

30. A polytetramethylene terephthalate shaped body, substantially as described in any one of the foregoing Examples 1 to 5.

31. A polytetramethylene terephthalate shaped body, substantially as described in any one of the foregoing Examples 6 to 13.

32. A polytetramethylene terephthalate shaped body, substantially as described in any one of the foregoing Examples 13 to 22.

33. A polytetramethylene terephthalate shaped body as claimed in Claim 1, substantially as described in the foregoing Example 23.

34. A process for the production of a stabilised polytetramethylene terephthalate shaped body as claimed in any one of Claims 1 to 29, which comprises continuously supplying to an extruder polytetramethylene terephthalate, triglycidyl isocyanurate and/or a bisoxazoline and optionally one or more further components is/are chosen from a said reinforcing filler, said impact resisting agents and said polymerised bromine-containing acrylate or methacrylate flame protecting agent, which component(s) is/are supplied separately from one another and separately from the polytetramethylene terephthalate, the triglycidyl isocyanurate and/or bisoxazoline and, if used, the phosphitic or phenolic antioxidant and/or  $Sb_2O_3$  being simultaneously supplied continuously and separately from one another to the extruder either together with the polytetramethylene terephthalate or one or more of said components or as materials supplied separately from any other constituent(s) of the body, and co-extruding said constituents in the extruder.

35. A process for the production of a stabilised polytetramethylene terephthalate shaped body, substantially as described in any one of the foregoing Examples.